N m Washington, D.C. 20231

Sir:

Transmitted herewith for filing is the Patent Application of:

Inventor(s): Fred R. Huege, Starr Curtis

For: COMPOSITION FOR ASPHALT ROOFING MATERIALS

**Enclosed are:** 

- X Patent Specification and Declaration
- X 10 sheets of drawing(s).
- X An assignment of the invention to Chemical Lime Company (includes Recordation Form Cover Sheet).
- A certified copy of a \_\_ application.
- Information Disclosure Statement, PTO 1449 and copies of references.

The filing fee has been calculated as shown below:

For	Number Filed	Number Extra	Rate	Fee
Basic Fee				\$690.00
Total Claims	36 - 20	16	x 18 =	\$288.00
Indep. Claims	4 - 3	1	x 78 =	\$ 78.00
MULTIPLE DEPENDEN	T CLAIM PRESENTED		x 260 =	\$

Total \$1,056.00

- X Enclosed please find a check in the amount of \$1,056.00 for the filing fee.
- X Enclosed please find a check in the amount of \$40.00 for the assignment fee.
- X The Commissioner is hereby authorized to charge payment of the following fees associated with this communication or credit any overpayment to Deposit Account 06-0580. A duplicate copy of this sheet is enclosed.
- X Any additional filing fees required under 37 CFR §1.16.
- X Any patent application processing fees under 37 CFR §1.17.

CERTIFICATE OF MAILING BY "EXPRESS MAIL" UNDER 37 CFR § 1.10

"Express Mail" mailing label number: EL139165025US

Date of Mailing: April 11, 2000

I hereby certify that the documents indicated above are being deposited with the United States Postal Service under 37 CFR 1.10 on the date indicated above and are addressed to Box Patent Applications, Assistant Commissioner for Patents, Washington, D.C. 20231 and mailed on the abone Date of Mailing with the above "Express Mail" mailing label hun

Christa King

(name of person mailing paper) SIGNATURE of person mailing paper of

Respectfully submitted,

Charles D. Gunter, Jr.

Registration No. 29,386 for FELSMAN, BRADLEY, VADEN, **GUNTER & DILLON, LLP** 201 Main Street, Suite 1600 Fort Worth, Texas 76102 Telephone (817) 332-8143

# SPECIFICATION

Docket No. 0438CG-54

## TO ALL WHOM IT MAY CONCERN:

BE IT KNOWN that we, Fred R. Huege, a resident of the state of Texas, and Starr Curtis, a resident of the state of Arizona, both citizens of the United States of America, have invented new and useful improvements in a

# COMPOSITION FOR ASPHALT ROOFING MATERIALS

of which the following is a specification:

"EXPRESS MAIL" NO. KEYBOARD (AIRBILL NUMBER)

I hereby certify that this paper or fee is being deposited with the United States Postal Service as "Express Mail Post Office to Addressee" service under 37 C.F.R. § 1.10 on the date indicated below and is addressed to the Assistant Commissioner for Patents, Washington, D.C. 20231.

Date of Deposit: 19-11-00

y: (Musica)

## BACKGROUND OF THE INVENTION

2

1

## 1. Field of the Invention:

4

This invention relates to asphaltic or bituminous 5 6 roofing materials and methods, and more particularly to the manufacture of such roofing materials to which 7 hydrated lime (Ca(OH)<sub>2</sub>, abbreviated as "HL") is added to strength, durability and 9 improve the antioxidant 10 qualities of the roofing material. The roofing materials may be shingles, rolls, or other materials to be placed 11 12 on roofs.

13

14

## 2. Description of the Prior Art:

15 16

17 Asphalt roofing shingles are manufactured by taking a continuous base sheet of organic felt or fibreglass 18 (generally, a web or form), saturating it in a base 19 asphalt, covering it with a coating asphalt, and then 20 embedding granules on the top side of the coated sheet. 21 22 The granules protect the asphalt from breaking down 23 through oxidization by ultra violet rays. Fillers such as 24 CaCO<sub>3</sub> and/or fly ash may also be used in the asphalt 25 composition. Finally, synthetic polymeric materials may also be added to the asphalt. The finished sheet is cut 26 27 into lanes and to a desired length of shingles.

28

More specifically, in the manufacture of roofing shingles or rolls, a heated asphaltic/filler blend is applied to a substrate web or substrate form, such as a glass fiber mat or a felt. The mat or felt is pre-shaped in the form that is desired for roofing purposes. After the form or web is impregnated with the asphaltic mix, a granular treatment may be applied to the hot asphalt surface and rolled or pressed into place. The coated web composition is then cooled so that it may be cut and

bundled as shingles, or formed into rolls.

7

9

10

11

12

13

14 15

16 17

18

19 20

21

22

23

6

The use of tar with HL to cover roofs is old in the art, as disclosed in U.S. Pat. No. 61,787 (disclosing first coating wooden shingles with HL, allowing it to dry, then coating the shingles with the tar, followed by sand). Asphaltic or bituminous materials as used in the roofing industry for pre-fabricated shingles or asphalt rolls are well known in the art, with the examples being described in U.S. Pat. No. 4,405,680 (disclosing a specific type of asphalt with glass filaments and method of manufacture), and 4,559,267 (disclosing a sealant bound to asphalt sheets). Prior to application to the substrate or web form, the asphalt is typically heated in an asphalt heater to a temperature of up to 500°F. heated asphalt is then blended with a filler that may or may not be chemically inert, the filler also having been preheated to a temperature necessary so as not to chill the mix and to facilitate blending with the asphalt.

2425

26

27

28 29

30

31

The choice filler has traditionally been based on considerations of availability, compatibility and cost. An inert filler material which has been preferred and used by many roofing plants is powder limestone (CaCO<sub>3</sub>) or dolomite (CaCO<sub>3</sub>·MgCO<sub>3</sub>), usually at a rate of about 40% to 70% by weight of the mix. Other materials may also be

blended with the asphalt, such as block and anti-block
polymers and thinners, as well known in the art.

3

Powder limestone often has been a filler of choice 4 as it is widely available at a relatively low cost, and 5 is compatible with the asphalt mix. 6 However, it is a poor conductor of heat when compared to fly ash. 7 relatively slow to heat, and therefore, in the mix, tends 8 to insulate the asphalt and retards the cooling of the 9 composite web or form. Further, CaCO3 is an active base 10 material, and it therefore tends to be acted upon by the 11 weak acid and the precipitation (acid rain) and is 12 believed to contribute to a shortened life of the roofing 13 material. More importantly, limestone fillers have been 14 documented as the cause of algae growth and discoloration 15 asphaltic shingled roofs. This is 16 undesirable since it decreases the life of the shingles, 17 and also lowers the aesthetic quality of the shingles. In 18 this regard, MgCO<sub>3</sub> may be a better filler for asphalt. 19

20

In any case, it is desirable to have present in the 21 asphalt shingle a substance that can counter these and 22 other detrimental effects of filler agents or the asphalt 23 itself. At the same time, it is desirable to improve the 24 durability of roofing material and increase its tear 25 strength. While HL has been recently disclosed in use as 26 a filler in asphalt for paving roads (U.S. Ser. No. 27 09/110,410, filed on July 6, 1998), HL has not been used 28 in asphalt shingles or other roofing materials. 29 the present invention discloses a roofing composition 30 31 that is an improvement on the prior art that incorporates 32 the advantages of HL.

#### SUMMARY OF THE INVENTION

2

3

5

6

1

One object of the present invention is to provide an improved roofing composition of shingles or asphalt roll having a low cost additive that imparts improved tear strength to the shingles, and is cost effective to use.

7

9

Another object of the present invention is to provide an asphalt shingle of improved strength that can be made with various types of asphalt.

11 12

13

14

15

16

17

18

19

20

21

22

23

24

25

26

27

10

These and other objects are achieved in an asphalt roofing composition in the form of a roll or a shinglelike structure in which a hot mixture of an asphaltic base and filler is applied to a substrate form, wherein the composition also comprises an amount of hydrated lime (HL, such as any alkaline earth metal hydroxide) in order to impart strength and durability to the composition. The composition contains HL between about 1-10%, and preferably between about 3-5%, of the asphalt by weight. The filler can be fly ash, CaCO<sub>3</sub>, MgCO<sub>2</sub>·CaCO<sub>3</sub>, MgCO<sub>3</sub>, or other suitable materials known in the art. In a typical embodiment of the invention, the HL is added directly to the asphaltic base of the composition either with the filler, or with filler added after mixing the asphalt and HL, or mixing the asphalt with the filler and then adding the HL.

28

29 Additional objects, features and advantages will be 30 apparent in the written description which follows.

1	BRIEF DESCRIPTION OF THE DRAWINGS
2	
3	Figure 1 is a graph of a SHRP parameter as a measure
4	of permanent deformation potential for different asphalt
5	binders with the addition of 20% HL by weight of asphalt
6	binder;
7	
8	Figure 2 is a graph of the change in viscosity of
9	one HMA composition as a function of reaction time and
10	blending time;
11	
12	Figure 3 is a graph similar to Figure 2, but showing
13	the results obtained with a second HMA composition;
14	
15	Figure 4 is a graph of fracture toughness for one
16	HMA composition with the addition of 20% by weight of HL;
17	
18	Figure 5 is a graph similar to Figure 4, but with a
19	second HMA composition;
20	
21	Figure 6 is a graph of accumulated shear deformation
22	for HMA mixes using two different bituminous binders;
23	
24	Figure 7 is a graph of controlled-stain fatigue life
25	comparing HMA's with and without the addition of HL;
26	
27	Figure 8 is a top view of a typical asphalt shingle;
28	
29	Figure 9 is a top view of a roof having asphalt roll
30	material placed below a series of asphalt shingles; and
31	

- 1 Figure 10 is a graph of the tear strength of the
- 2 shingles of the invention when compared to traditional
- 3 (control) shingles.

#### DETAILED DESCRIPTION OF THE INVENTION

2

1

The following abbreviations are used throughout
the specification: SHRP is Strategic Highway Research
Program; HL is hydrated lime (Ca(OH)<sub>2</sub>, Mg(OH)<sub>2</sub> or
Ca(OH)<sub>2</sub>·Mg(OH)<sub>2</sub>); HMA is hot mix asphalt; and IDT is
Indirect Tensile. Other abbreviations are defined as
they are used.

9

The present invention is directed to improvements in 10 roofing materials and similar 11 compositions in which a lime component, preferably HL, is 12 added directly to the asphalt or asphalt in one 13 14 embodiment, or first to the filler and then to the in another embodiment. In vet 15 asphalt embodiment, the HL is added to the mixture of filler and 16 asphalt. Hereinafter, the terms "bitumen" and "asphalt" 17 are used interchangeably. Further, the term HL is used 18 to refer in general to any alkaline earth metal hydroxide 19 such as Mq(OH)<sub>2</sub> and Ca(OH)<sub>2</sub>, or mineral mixture thereof 20 (generally, Ca(OH)2 Mg(OH)2). In the disclosure which 21 follows, the term "quicklime" refers to alkaline earth 22 metal oxides such as CaO, while the use of HL refers to 23 alkaline earth metal hydroxides such as Ca(OH)2. 24

25

26

27

28 29

30

31

32

In the production of HL, limestone or calcium carbonate is first heated to remove carbon dioxide. The remaining CaO is a very active chemical. To improve the handling characteristics of the quicklime, a controlled amount of water is added to form HL. Adding a HL component to the aggregate or filler (e.g., rock, sand, fly ash, limestone) in asphaltic compositions is done in

the present invention with the intention of improving the 1 bond between the aggregate/filler, fiber glass matte or 2 other substrate form, and asphalt, especially in the 3 presence of water which has a stronger affinity for the 4 aggregate than the asphalt does. This in turn improves 5 the tear strength of the shingle-like structure or 6 roofing material. Hydrated lime added to the aggregate 7 is an effective antistripping agent and has been 8 considered to have ancillary positive effects on the 9 asphalt mixture. 10

11 12

13

14

15

16

17

18

19

20

21 22

23

2425

26

27

The mechanism by which HL improves aggregate-asphalt adhesion and moisture sensitivity when the HL is added directly to aggregate is reasonably well understood although some arguments still exist as to the mechanisms responsible. It is theorized that the lime decreases the interfacial tension between the asphalt and water, thus resulting in good adhesion. It is also thought that the HL improves the stripping resistance by interacting with the carboxylic acids in the asphalt. This interaction forms insoluble products that are readily adsorbed onto the surface of the aggregate or filler, or in the specific case of roofing materials, the substrate form or web used to make the shingle-like structures or rolls. Some studies indicate that strong adsorption of calcium onto mineral aggregate surfaces may contribute to bonding of asphalt cements with the aggregate or filler.

28

The following data demonstrates that HL added directly to asphalt has a multi-functional effect. The effect which is achieved is more than simply that of an antistrip additive. Hydrated lime was added directly to

five different asphalts (denoted AAB, AD, AAF, AAG and 1 2 AAM) which represent the range of asphalts that would reasonably be encountered in the United States and 3 throughout most of the world, as discussed in Table 1. 4 5 Each of the selected asphalts represents a wide variety of asphalt chemical and physical properties. 6 research in the asphalt study concentrated on using 7 8 testing techniques that are now being accepted by the industry as part of the Strategic Highway Research 9 Program's (SHRP) Superpave protocol. However, some non-10 traditional tests were also performed. The testing 11 protocol is given below in Table 1. Although these data 12 apply to asphalt compositions for road use, they also 13 equally apply in general to the use of asphalt/HL 14 15 compositions in any conditions where there is exposure to weather and physical stresses. These results, along with 16 those discussed in Figure 10 below, show that the 17 18 asphaltic shingle composition of the present invention has improved characteristics relative to typical asphalt 19 20 shingles, the HL having unexpected benefits.

- 10 -

Table 1. Tests performed upon various bituminous compositions.

	Parameters	
Test	Measured	Purpose of Test
Series I - Investigation of low temperature performance  IDT - Performed at three low temperatures for one hour to provide low temperature creep compliance on mixtures subject to aging (loose mix and compacted mix) according to Superpave protocol	Creep compliance versus time of loading - ultimate compliance, rate of change in compliance	Assess low temperature fracture properties  (2 replicates for each mixture system - 18 samples)
IDT - Tensile strength at three low temperatures on mixtures subjected to aging as described above (AAMAS protocol)	Stress and strain at failure	Assess low temperature fracture properties (18 samples)
Series II - Investigation of intermediate temperature performance  IDT - creep and tensile strength at intermediate temperature (20°C) to assess fracture properties (AAMAS protocol)	Creep compliance versus time of loading - ultimate compliance, rate of change in compliance	Assess intermediate temperature fracture fatigue properties  (36 samples)
Series III - Investigation of moisture resistance Perform AASHTO T-283	Retained tensile strength	Assess effect of HL on moisture resistance (18 samples)
SERIES IV - Investigation of high temperature performance  Compressive creep performed at 60°C one hour to assess permanent deformation potential (AAMAS protocol)	Creep compliance versus time of loading - ultimate compliance, rate of change in compliance	Assess effect of HL on high temperature rutting (6 samples)

2 3	Test	Parameters Measured	Purpose of Test
4 5 6 7 8 9 10 11	Repeated load (axial loading) permanent deformation testing at 60°C	Ultimate accumulated strain, rate of accumulated strain and slope of steady state region	Assess susceptibility of permanent deformation and the effect of HL (6 samples)
13 14 15 16	Repeated shear permanent deformation testing at 60°C	Same as above	Same as above

The following summary of the experimental work is 2 divided into three sections: high temperature rheology, 3 low temperature rheology and intermediate temperature 4 rheology. At high temperatures, asphalt becomes soft and 5 susceptible to shoving and rutting when used in roadways, 6 and creeping or deformation when used as roofing 7 materials. The tests performed evaluated the ability of 8 the asphalt to withstand the stresses induced in high 9 temperature environments. At low temperatures, asphalt 10 becomes hard and susceptible to fracture. 11 particularly true for asphalt mixtures that have become 12 embrittled due to aging. The tests performed at low 13 temperatures evaluate the ability of the asphalt to 14 withstand load-induced and environmentally 15 stresses at low temperatures. Load-induced fatigue 16 typically occurs at low and intermediate 17 cracking The test performed at intermediate or temperatures. 18 average temperatures assess the ability of the asphalt to 19 withstand fatigue at average or nominal temperatures. The 20 tests were conducted by reacting the asphalts in mass 21 with the HL in closed containers in accordance with the 22 previously enunciated testing protocols. 23

24

Evaluation of the Effects of HL on High Temperature Rheology. Hydrated lime added directly to the asphalt in selected ranges from about 10% to about 20% by weight, based on the total weight of asphalt binder produces several high temperature rheology effects which can be summarized as follows:

1 Hydrated lime added to asphalts has a very positive 2 filler effect. This effect substantially improves high temperature rheological parameters which relate to 3 resistance to permanent deformation. Figure 1 shows how 4 20% HL by weight asphalt binder dramatically changes the 5 SHRP parameter  $G^*/\sin \delta$  which is related to permanent 6 A high  $G^*/\sin \delta$  results in 7 deformation potential. 8 reduced permanent deformation potential. Somewhere between 10% and 20% HL by weight asphalt binder is 9 10 required to provide the desired high temperature rheological changes. In the HL containing shingles, only 11 about 1 to 10% HL by total weight of asphaltic/filler 12 13 composition is required to effectuate an improvement in tear strength and antioxidant properties. 14

15

The high temperature rheology of HL-filled asphalts is dependent on the time and temperature of blending of HL with the asphalt. The process is asphalt specific. This finding demonstrates that the interaction between HL and asphalt is likely not simply physical but a chemical interaction may also exist.

22

23 Figures 2 and 3 illustrate the effect of reaction time at 149°C on HL in asphalt AAD to reaction time of 24 25 longer than five minutes. However, asphalt AAM requires 26 a reaction time of about 40 minutes to achieve viscosity 27 equilibrium. This indicates a physio-chemical interaction unique to specific binders. 28 Note that the 29 untreated asphalts are unaffected by reaction time. Since the asphalts were reacted in mass in closed 30 containers, oxidative aging should not be a factor. 31 32 However, the HL, when added to the asphalt in roofing materials, will decrease oxidative aging and thus improve
the performance of the shingles or roll materials.

Evaluation of the Effects of HL on Low Temperature
Rheology. The findings with regard to low temperature
rheology are summarized as follows:

Hydrated 8 lime increases the low temperature stiffness of asphalts indicating that they are more 9 10 susceptible to low temperature fracture. However, HL added at rates of 12.5% by weight of asphalt and below 11 has a small effect on low temperature stiffness and does 12 not significantly affect the slope of the stiffness 13 versus time of loading curve determined using the low 14 temperature Bending Beam Rheometer test. SHRP research 15 indicates that the slope is more important. Thus, adding 16 1-10% HL to the asphalt composition will also improve the 17 18 properties of the roofing materials.

19 20

21

22

23

24

25

26

To evaluate whether the stiffness increase at low temperature due to HL addition is important, low temperature fracture tests were performed. Hydrated lime substantially improves low temperature fracture toughness. The improved fracture toughness and minimal effect on the slope of the stiffness versus time of loading curve indicates improved low temperature crack resistance despite the increased stiffness.

28

27

Figures 4 and 5 illustrate the effect of HL in improving fracture toughness.

The improved low temperature properties are due to a synergistic effect of reduction in the effect of oxidative aging (as all samples are aged to simulate pavement aging before testing) and crack pinning, a phenomenon of energy dissipation due to microcrack interception by the dispersion of HL particles in the asphalt.

8

Evaluation of Effects of HL on Intermediate Temperature 9 The filler effect of HL is obvious at all 10 Rheology. However, at low temperatures 11 temperatures. stiffening effect was proven to be more than compensated 12 for by the improvement in fracture toughness. 13 generally recognized accepted binder tests are available 14 by which to evaluate intermediate temperature fatigue 15 susceptibility. Therefore, the following mixture tests 16 direct tensile fatigue tests and microcrack 17 These tests provided favorable results healing tests. 18 which are discussed in the mixture section. 19

20

HL in Asphalt -- Effects on Mixture Properties. Hydrated 21 lime was added to two asphalts with very different 22 chemical and physical properties. These asphalts are 23 Mixtures with Watsonville designated AAD and AAM. 24 granite aggregate and 5.05% asphalt by total weight of 25 the mixture were subject to two types of mixture tests: 26 repeated shear permanent deformation testing and direct 27 28 tensile fatique testing. The repeated stress, permanent deformation testing was performed to assess rutting 29 potential in the mixtures tested. The direct tensile 30 fatigue testing was performed to assess the effect of 31 lime on the potential of the mixture to develop fatigue 32

1 cracking. These are two of the dominant distress

2 mechanisms in hot mix asphalt pavements and are

3 responsible for the vast majority of pavement damage and

4 deterioration.

5

Results of Permanent Deformation Testing. The repeated shear permanent deformation testing was performed at 40°C. The testing was performed using a testing protocol developed in the SHRP research program to simulate the stress state that an asphalt mixture is subjected to under a moving wheel load. During the testing sequence the mixture is subjected to a constant ratio of axial

stress and repeated shearing stresses.

14

13

Tests were performed on HMA mixtures prepared with 15 four different asphalt binders with and without HL as 16 follows: AAD, AAD 12.5% HL, AAM and AAM with 12.5% HL. 17 Three identical samples were prepared for each mixture 18 and the mixtures were subjected to a 20,000 lbs. load 19 application. The tests revealed that the addition of HL 20 reduced the level of permanent deformation on average 21 about 300% (Figure 6), based on values of ultimate 22 permanent strain after 20,000 cycles. The data were 23 considerably variable, however. Although the above tests 24 were performed on mixtures of asphalt and granite 25 aggregate, the same or similar results are expected with 26 glass filaments and/or fly ash, or the substrate forms 27 used to make the shingles or roofing rolls of the 28 invention, as they have the common property of being 29 siliceous and/or carbonaceous. 30

Results of Direct Tensile Fatigue Testing. The purpose 1 of direct tensile fatigue testing was to assess the 2 resistance of asphalt mixtures to load-induced 3 (controlled-strain) fatigue testing at intermediate (or 4 average annual) pavement and exterior temperatures that 5 shingles will be exposed to. Identical mixtures of 6 Watsonville granite and 5.0% asphalt (by total weight of 7 the mixture) were prepared with asphalt binders with and 8 without HL as follows: AAD, AAD with 12.0% HL, AAM and 9 AAM with 12.5% HL. Analysis of the results of 10 controlled-strain fatique testing demonstrated two 11 First, at a given level of stiffness, the 12 findings. addition of HL improved fatigue life. Second, the 13 recovery of dissipated energy (responsible for crack 14 healing) after rest periods is enhanced by the addition 15 of HL for mixtures subject to age hardening. For a given 16 design stiffness and for a mixtures subject to age 17 hardening, the addition of HL appears to enhance the 18 resistance to fatigue cracking. 19

2021

22

23

Figure 7 illustrates typical fatigue results where cycles to failure (Nf) are compared for untreated and HL treated mixtures at various mixture stiffness.

24

An invention has been shown with several advantages.

HL is an effective multi-functional additive which is
effective in improving the high temperature performance
of hot mix asphalt.

29

30 Uniaxial tensile controlled strain fatigue tests, 31 performed on mixtures with and without the addition of HL 32 added directly to the binder, demonstrate that the lime 1 addition improves the fatigue life of the mixture

2 (resistance to cracking) when mixtures are compared at a

3 common level of stiffness.

4

5 Shingles, shingle-like structures which come in various forms, or asphalt rolls used for roofing 6 typically are made from glass impregnated mats or 7 substrate forms, the asphalt and fillers, etc. being 8 bound and formed around the mat or form. The shingles 9 can be as shown in Figure 8, wherein asphalt shingle 10 10 having an adhesive strip 12 is shown. A number of 11 shingles 10 are placed upon a roof 16 as in Figure 9, 12 wherein sheet material from a roll of asphalt material 14 13 is first placed on the roof underneath the layered 14 The roll comprises a rolled sheet of shingles 10. 15 asphaltic material, usually formed around a substrate web 16 or form, the layer of material placed directly in contact 17 with the wood roofing material prior to addition of the 18 shingles as in Figure 8. Further, various polymers can 19 be added to the asphalt along with the HL of the 20 invention, such as disclosed in U.S. Pat. No. 4,405,680. 21

22

composition include Typical fillers for the 23 limestone and/or dolomite dust and glass fibers of 24 25 various sizes and lengths, sand, rock (of various mineral composition), and other substantially siliceous materials 26 in ground and/or powdered form. The asphalt utilized in 27 the asphalt composition of the present invention is 28 typical of the industry. An asphalt of this type 29 typically has a softening point of between, for example, 30 190°F and 240°F and a penetration at 77°F between, for 31 32 example, 14 dmm and 25 dmm (dmm is tenths of a

millimeter). In saturating the substrate form, the asphalt is maintained in a molten state, preferably at a temperature any where between 350°F and 450°F. At this temperature and without any fillers or additives, the molten asphalt has a viscosity and Saybolt furol seconds of 100 and 300.

7

The physical properties of the asphalt, as recited 8 herein, are for exemplary purposes only. Any asphalt 9 which functions in the manner to be described herein may 10 be utilized, and in fact, may be readily provided by 11 those skilled in the art. In this regard, the saturating 12 or coating temperature of the molten asphalt, or the 13 operating temperature as it is commonly called, will 14 depend in part on the particular asphalt used and in part 15 on other ingredients in the overall composition. 16 event, the temperature of the asphalt 17 sufficiently high to readily saturate or coat the 18 substrate form with the asphalt composition, yet it 19 should not be maintained at a temperature higher than 20 necessary. This is, of course, because a large amount of 21 energy is required to maintain the composition in its 22 molten state. 23

24

25

26

27

28 29

30

31

32

The asphalt composition of the present invention preferably includes between about 30% and 50% asphalt by weight of the total composition. When less than approximately 30% is provided, the asphalt does not satisfactorily fulfill its intended purpose, that is, it does not satisfactorily provide the ultimately produced shingle or roll with adequate physical characteristics. In addition, it tends to be too viscous at the preferred

saturating temperatures and thus increases creep. On the other hand, providing the composition with more than 50% asphalt is not necessary and, taking into account costs considerations, is not preferable. In this regard, to extend the asphalt, a suitable conventional filler, such as for example, limestone and other mineral filler is added thereto.

8

10

11

12

13

14

15

16

17

18

19

20

21 22

The mineral filler is dispersed throughout the asphalt by conventional means. For example, mechanical agitation, when the asphalt is in its molten state, preferably at this saturating temperature. approximately 45% and 55% mineral filler, by way of the total composition, is preferably utilized. The exact percentage of mineral filler provided will be dictated by the amount of asphalt and the amount of glass in the form of glass fiber bundles which are utilized in the especially when these are the composition, ingredients comprising the composition. Of course, the filler must not be of a type or an amount which will prevent saturation of the base sheet at any reasonable saturating temperature.

23

24

25

26 27

28

29

30

31

32

There are several principle methods of mixing the composition of the invention. The first is to add the HL to the molten asphalt. The second is to add the HL to the filler first, mixing or agitating it thoroughly first, with or without excess water, then adding the molten asphalt. A third method is to first mix the asphalt and filler, then add the HL to the mixture. In either case, the HL is added to between 1% and 10% of the added asphalt. Preferably, the HL is added to an amount

1 between 3% and 5% of the weight of the asphalt. Hydrated

2 lime is added as a powder. The asphalt/HL and/or

- 3 filler/HL mixtures are typically agitated to achieve an
- 4 uniform distribution of the lime. This can be done with
- 5 a pug mill, however, in some cases vigorous mixing is not
- 6 necessary.

7

In one embodiment, the HL is added directly to the 8 filler first as a dry powder, both ground and mixed to 9 form a homogeneous mixture. The HL can be added prior to 10 or after grinding the filler material. 11 In yet another embodiment, CaO (or CaO MgO) is added to wet or damp 12 rock, thus being hydrated in a reaction between the CaO 13 and H<sub>2</sub>O to form Ca(OH)<sub>2</sub> (or Ca(OH)<sub>2</sub>·Mg(OH)<sub>2</sub>). The reaction 14 mixture is then ground to the desired particle size. 15 yet another embodiment, CaO (or CaO MgO) or HL slurry is 16 added to the rock prior to grinding to the desired 17

19

18

particle size.

The HL can also be mixed with the asphalt as the asphalt is heated to make the mixing easier. The temperature is dependent upon the type of asphalt, as discussed above, and its viscosity.

24

As stated above, in accordance with the present 25 invention, a small percentage of glass in the form of 26 glass fiber bundles is added to the asphalt filler 27 These glass fiber bundles are dispersed 28 mixture. 29 throughout this mixture and could be dispersed throughout the asphalt prior to the addition of the filler but in 30 any case, are added while the asphalt is in its molten 31 state, preferably at its saturating temperature. In this 32

regard, the glass may be dispersed in the asphalt by, for example, mechanical agitation.

3

As stated previously, the asphalt-filler mixture, 4 when maintained at the saturating temperature between 5 350°F and 450°F, will have a sufficiently low viscosity 6 so as to permit easy saturation of the base sheet. While 7 the addition of the glass fiber bundles to this mixture 8 will increase the viscosity slightly, the amount and type 9 of bundle selected must be such that the overall 10 the saturating temperature has 11 composition at sufficiently viscosity level to permit easy saturation of 12 13 the base sheet.

14

The exact type of glass fiber mat which could be 15 used may vary and could also be readily determined by 16 those skilled in the art in view of the teaching of the 17 present invention. However, those which have been found 18 to be acceptable are between approximately 1/8 and 1/2 in 19 length, including between 100 and 800 per bundle and 20 having a filament diameter of, for example, 13 to 18 21 The binder utilized in holder the micro 22 micrometers. filaments together must be, of course, one of which will 23 continue to hold the bundles together, to at least to a 24 substantial degree, and the saturating temperature of the 25 asphalt, even though the asphalt is mildly agitated to 26 disperse the glass bundles. It must also be one which by 27 28 melting, dissolving or any other way, allows the fiber bundles to defilamentize to a large extent 29 substantially higher asphalt temperatures, for example, 30 at temperatures in excess of 700°F. Limestone (CaCO<sub>3</sub>), 31

sand, fly ash, and other siliceous materials are typical binders used each alone or in some combination.

3

In one embodiment of the present invention, the 4 shingles are manufactured by first providing a fiberglass 5 mat of a preformed shape and size. Hot asphalt having 6 the filler and HL is then applied to the top of the mat 7 to the desired thickness. The asphalt is then allowed to 8 dry. Next, the uncoated side of the mat is exposed, and 9 hot asphalt is then applied to the uncoated surface to 10 the desired thickness. This is then allowed to dry. 11 a finished product, sand or other decorative material may 12 be added to the surface prior to drying to adhere the 13 sand or other material the surface of the shingle. 14

15

The shingles of the present invention have the 16 unexpected advantage of having a greater tear strength 17 than traditional asphalt shingles not using HL. The data 18 in Figure 10 highlight this aspect of the present 19 invention, wherein control samples (filled circles) of 20 shingles that do not have HL were compared with the HL 21 containing shingles (closed squares) of the present 22 invention. Shingles of various thickness (in fractions 23 of an inch) were compared and the tear strength of each 24 compared to one another. The data for the HL shingles 25 use a composition of 3% HL by weight of asphalt. 26 line 101 is a best-fit line through the data for the HL 27 shingles of the invention, while the line 103 is the 28 best-fit line through the data for the control. 29

30

The increased tear strength of the HL shingles of the present invention is an advantage over prior shingles. The shingles of the present invention are more durable than prior shingles, and at a minimal added cost as HL is a low cost additive. The low cost is an advantage when compared to other asphalt shingles using polymeric materials as additives to improve strength.

6

While the invention has been shown in only one of its forms, it is not thus limited but is susceptible to various changes and modifications without departing from the spirit thereof.

1	What	is claimed is:
2		
3	1.	An asphalt roofing composition in the form of a roll
4		or a shingle-like structure in which a hot mixture
5		of an asphaltic base and filler is applied to a
6		substrate form, wherein the composition also
7		comprises an amount of an alkaline earth metal
8		hydroxide in order to impart strength and durability
9		to the composition.
10		
11	2.	The composition of Claim 1, wherein the alkaline
12		earth metal hydroxide is selected from a group
13		consisting of $Ca(OH)_2$ , $Mg(OH)_2$ , and $Ca(OH)_2 \cdot Mg(OH)_2$ .
14		
15	3.	The composition of Claim 1, wherein the alkaline
16		earth metal hydroxide is between about 1-10% by
17		weight of asphalt.
18		
19	4.	The composition of Claim 1, wherein the alkaline
20		earth metal hydroxide is between about 3-5% by
21		weight of asphalt.
22	•	
23	5.	The composition of Claim 1, wherein the filler is
24		fly ash.
25		
26	6.	The composition of Claim 1, wherein the filler is
27		CaCO <sub>3</sub> .
28		
29	7.	The composition of Claim 1, wherein the filler is
30		MgCO <sub>3</sub> or MgCO <sub>2</sub> ·CaCO <sub>3</sub> .

1	8.	The composition of Claim 1, wherein the alkaline
2		earth metal hydroxide is first added directly to the
3		asphaltic base of the composition.
4		
5	9.	The composition of Claim 1, wherein the alkaline
6		earth metal hydroxide is first added directly to the
7		filler of the composition.
8		
9	10.	The composition of Claim 1, wherein the alkaline
10		earth metal is added first to the filler then to the
11		asphaltic base of the composition.
12		
13	11.	The composition of Claim 1, wherein the composition
14		is between about 30% to 60% asphalt by weight.
15		
16	12.	An asphalt roofing composition in the form of a roll
17		or a shingle-like structure in which a hot mixture
18		of an asphaltic base, filler, and water is applied
19		to a substrate form, wherein the composition also
20		comprises an amount of an alkaline earth metal oxide
21		in order to impart strength and durability to the
22		composition, the metal oxide reacting with water in
23		the filler to produce the corresponding metal
24		hydroxide.
25		
26	13.	The composition of Claim 12, wherein the alkaline
27		earth metal oxide is selected from a group
28		consisting of CaO, MgO, and CaO·MgO.
29		
30	14.	The composition of Claim 12, wherein the alkaline

earth metal oxide is between about 1-10% by weight

of asphalt.

31

1		
2	15.	The composition of Claim 12, wherein the alkaline
3		earth metal oxide is between about 3-5% by weight of
4		asphalt.
5		
6	16.	The composition of Claim 12, wherein the filler is
7		fly ash.
8		
9	17.	The composition of Claim 12, wherein the filler is
10		CaCO <sub>3</sub> .
11		
12	18.	The composition of Claim 12, wherein the filler is
13		MgCO₃ or MgCO₂·CaCO₃.
14		
15	19.	The composition of Claim 12, wherein the alkaline
16		earth metal oxide is first added directly to the
17		asphaltic base of the composition.
18		
19	20.	The composition of Claim 12, wherein the alkaline
20		earth metal oxide is added first to the filler with
21		water, the oxide and water thus reacting to form the
22		corresponding hydroxide, the hydroxide and filler
23		then being added to the asphaltic base of the
24		composition.
25		
26	21.	The composition of Claim 12, wherein the composition
27		is between about 30% to 60% asphalt by weight.
28		
29	22.	A method of forming an asphalt roofing composition
30		in the form of a roll or a shingle-like structure,
31		the method comprising:

1		heating an amount of asphalt;
2		
3		providing a desired amount of an alkaline earth
4		metal hydroxide;
5		
6		providing a filler;
7		
8		combining the asphalt, metal hydroxide, and filler
9		to form the composition; and
10		
11		placing the composition onto a substrate form and
12		allowing the second hot mixture to cool around the
13		substrate form.
14		
15	23.	The method of Claim 22, wherein the hot asphalt and
16		the metal hydroxide are first mixed to form a
17		mixture, and the filler is then added to form the
18		composition.
19		
20	24.	The method of Claim 22, wherein the metal hydroxide
21		and filler are first mixed to form a mixture, and
22		the hot asphalt is then added to form the
23		composition.
24		
25	25.	The method of Claim 22, wherein the alkaline earth
26		metal hydroxide is selected from a group consisting
27		of $Ca(OH)_2$ , $Mg(OH)_2$ , and $Ca(OH)_2$ ·Mg(OH) <sub>2</sub> .
28		
29	26.	The method of Claim 22, wherein the alkaline earth
30		metal hydroxide is between about 1-10% by weight of
31		asphalt.

1	27.	The method of Claim 22, wherein the alkaline earth
2		metal hydroxide is between about 3-5% by weight of
3		asphalt.
4		
5	28.	The method of Claim 22, wherein the composition is
6		between about 30% to 60% asphalt by weight.
7		
8	29.	The method of Claim 22, wherein the substrate form
9		is a fiberglass mat.
LO		
L1	30.	A method of forming an asphalt roofing composition
L2		in the form of a roll or a shingle-like structure,
L3		the method comprising:
L4		
L5		heating an amount of asphalt;
L6		
L 7		providing a desired amount of an alkaline earth
L8		metal oxide;
19		
20		providing a filler and water;
21		
22		combining the asphalt, metal oxide, water and filler
23		to form the composition; and
24		
25		placing the composition onto a substrate form and
26		allowing the second hot mixture to cool around the
27		substrate form.
28		
29	31.	The method of Claim 30, wherein the metal hydroxide
30		and filler are first mixed with water to form a
31		mixture, and the hot asphalt then being added to
32		form the composition.

15

3		CaO, MgO, and CaO·MgO.
4		
5	33.	The method of Claim 30, wherein the alkaline earth
6		metal oxide is between about 1-10% by weight of
7		asphalt.
8		
9	34.	The method of Claim 30, wherein the alkaline earth
10		metal oxide is between about 3-5% by weight of
11		asphalt.
12		

32. The method of Claim 30, wherein the alkaline earth

metal oxide is selected from a group consisting of

13 35. The method of Claim 30, wherein the composition is 14 between about 30% to 60% asphalt by weight.

16 36. The method of Claim 30, wherein the substrate form
17 is a fiberglass mat.

# ABSTRACT OF THE DISCLOSURE

2

3	An asphalt roofing composition is provided in the
4	form of a roll or a shingle in which a hot mixture of an
5	asphaltic base and filler is applied to a substrate form,
6	wherein the composition also comprises an amount of
7	${\rm Ca(OH)_{2}}$ (HL) in order to impart strength and durability
8	to the composition. The composition contains HL between
9	about 1-10%, and preferably between about 3-5%, of the
10	total composition by weight. The filler can be fly ash,
11	CaCO <sub>3</sub> , MgCO <sub>2</sub> ·CaCO <sub>3</sub> , MgCO <sub>3</sub> , or other suitable materials
12	known in the art. In a typical embodiment of the
13	invention, the HL is added directly to the asphaltic base
14	of the composition either with the filler, or with filler
15	added after mixing the asphalt and HL.

Effect of the addition of 20% HL by weight of bitumen on G\* sin delta at 70C.

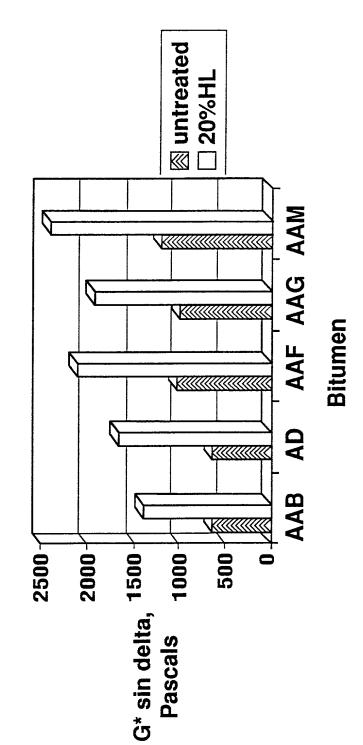


FIG. 1

Change in viscosity of AAD and HL as a function of reaction time and blending time.

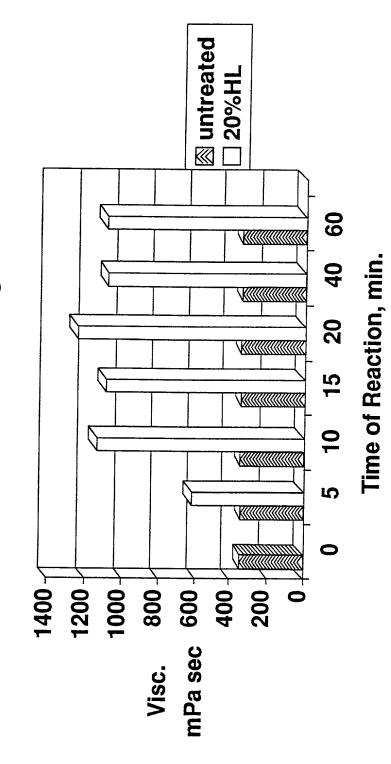
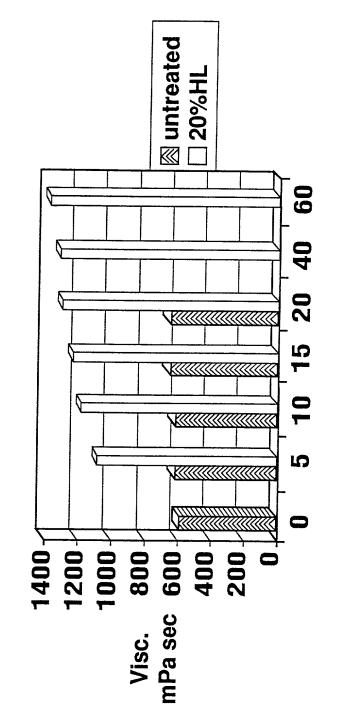


FIG. 2

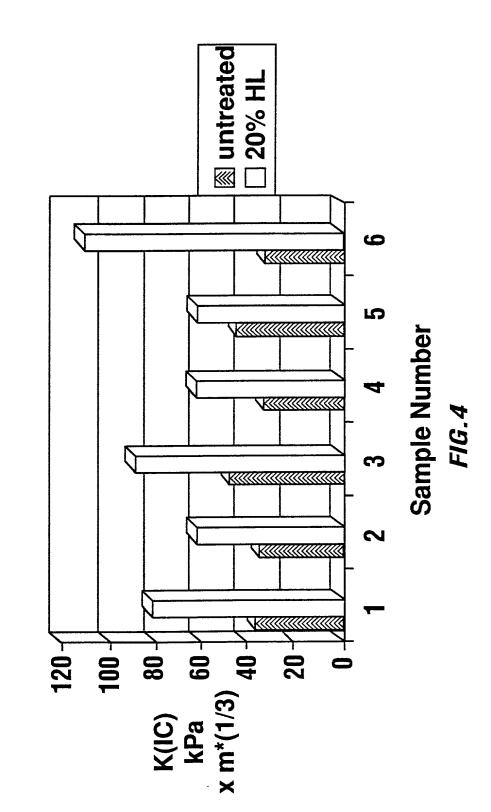
Change in viscosity of AAM and HL as a function of reaction time and blending time



Time of Reaction, min.

FIG. 3

Fracture toughness of AAD wih 20% HL at - 15C.



Effect of 20% HL on fracture toughness of AAM at -15C.

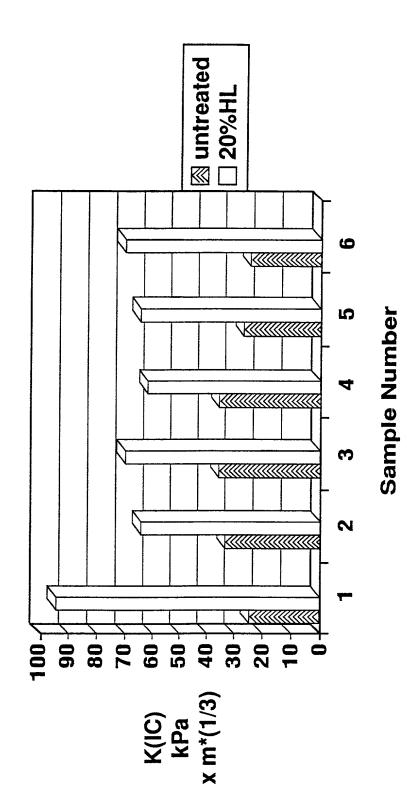
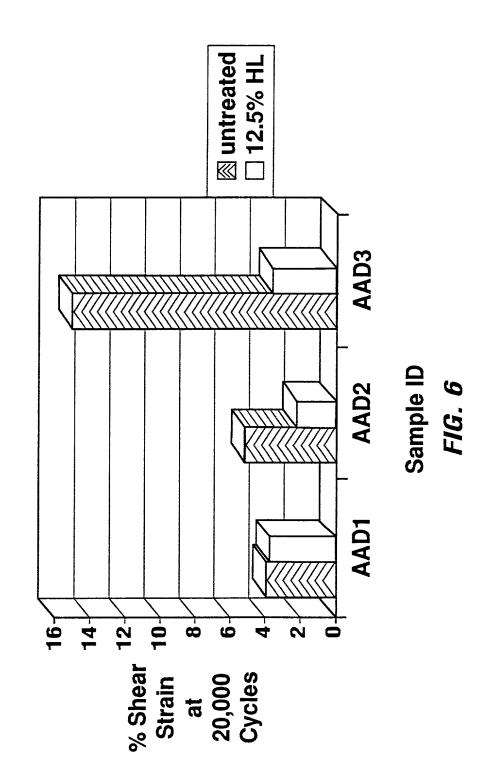


FIG. 5

Effect of HL on accumulated shear deformation for asphalt mixes using binders AAD and AAM.



Effect of HL on controlled-strain fatigue life based on regression analysis of 21 mixtures.

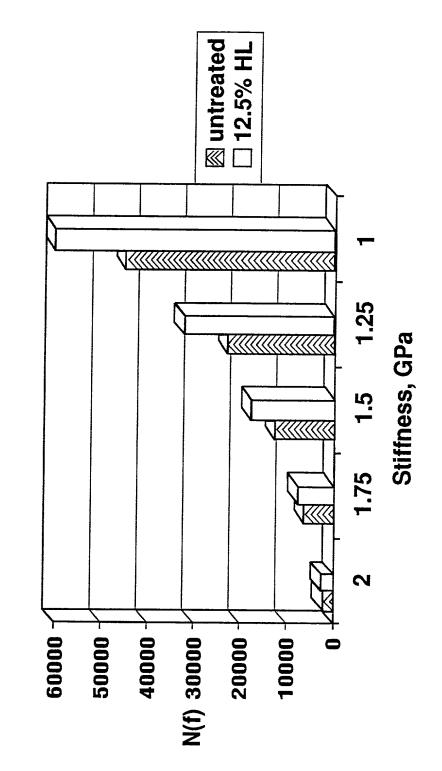
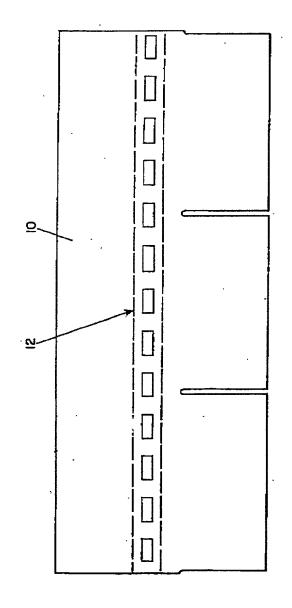


FIG. 7



7.00

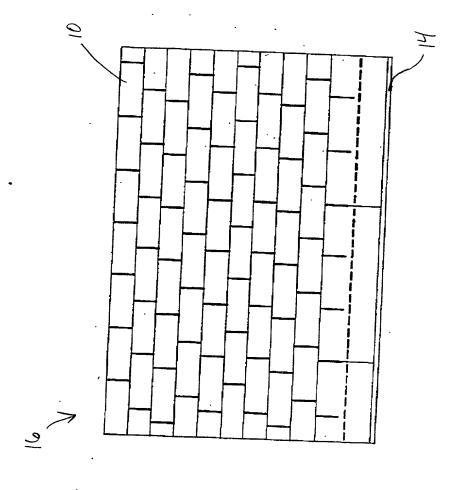
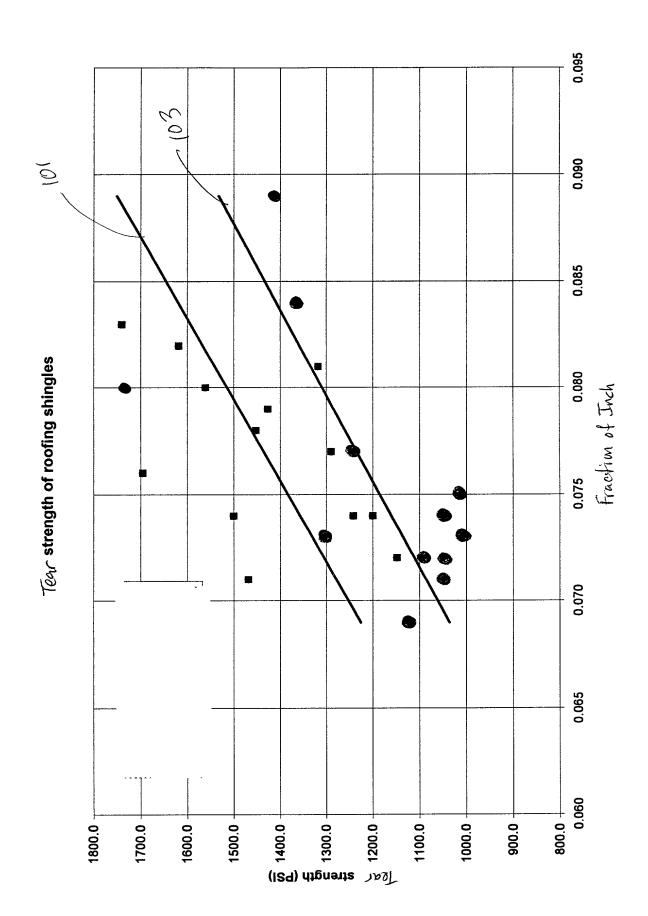


Fig. 9



#### DECLARATION FOR PATENT APPLICATION

As a below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name,

I believe I am an original, first, and joint inventor of the subject matter which is claimed and for which a patent is sought on the invention entitled

#### COMPOSITION FOR ASPHALT ROOFING MATERIALS

the specification of which is attached hereto.

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, Section 1.56.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

I hereby appoint James E. Bradley, Reg. No. 27,536; Charles D. Gunter, Jr., Reg. No. 29,386; Andrew J. Dillon, Reg. No. 29,634; Max Ciccarelli, Reg. No. 39,454; and Jack V. Musgrove, Reg. No. 31,986, to prosecute this application and to transact all business in the U.S. Patent and Trademark Office in connection therewith.

Please send all correspondence to:

Please send all correspondence to:

Charles D. Gunter, Jr.

Reg. No. 29,386

Felsman, Bradley, Vaden, Gunter & Dillon, LLP
201 Main Street, Suite 1600

Fort Worth, Texas 76102-3105

(817) 332-8143

Inventor's Signature: Told (Speed
Full Name of Sole or First Inventor: Fred R. Huege
Date of Signature: 3/20/00
Residence Address: 6403 Emerald Dr., Colleyville, TX 76034
Citizenship: USA
Post Office Address: same
Inventor's Signature: N. Stars Curtos
Full Name of Second Joint Inventor: Starr Curtis
Date of Signature: $4/4/2000$
Residence Address: 5838 North 42nd Street, Phoenix, AZ 85018
Citizenship: USA
Post Office Address: same